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THE PRIVILEGES AND RESPONSIBILITIES OF THE CHEMICAL ANALYST[1]

IN his presidential address on "The Dignity of Analytical Work," delivered before the American Chemical Society in 1897, Dr. Charles B. Dudley directed attention to the claims which may be asserted in behalf of analytical chemistry as an important and dignified branch of chemical science, and the necessity for intelligent, careful work on the part of even the routine analyst. From a somewhat different standpoint, Dr. W. F. Hillebrand, of international reputation as an analyst, presented at the Philadelphia meeting of the Society, in 1904, a critical review entitled "Some Thoughts on the Present Conditions of Analytical Chemistry," which was based upon his experience as chairman of committees intrusted with the formulation of standard methods of analysis and the investigation of the causes for the remarkable variation in the results of different analysts examining a given sample of material. He sounded a definite and clear note of warning with respect to the prevalence of a lack of care and intelligence, and ascribed the conditions, in part at least, to faulty training in our educational institutions. Within the past month there has appeared an interesting brochure from the pen of Dr. Wilhelm Böttger, professor at the University of Leipzig, entitled "Der Stand und Wege der Analytischen Chemie," in which he criticizes the too empirical nature of much of the analytical

[1] Read at a joint session of the American Chemical Society and Section C of the American Association for the Advancement of Science at Washington, December 27, 1911.

practise of the day and makes valuable suggestions respecting the way out of these conditions.

Dr. Dudley's plea was authoritative and convincing, and attracted much interest when it was made public. Dr. Hillebrand spoke from a fullness of experience and with an earnestness which made his criticism strike home. Dr. Böttger writes from the viewpoint of one who is at home in the analytical field and has also thoroughly familiarized himself with the advances of chemical science in other lines which are closely related to the work of the analyst. Is there, then, an excuse for a fresh homily on the status of analytical chemistry? May I say frankly that, had the pamphlet from Dr. Böttger reached me before the title of this address had been announced and its general outline prepared, I should have been inclined to answer this query in the negative, and may I confess that, since Dr. Böttger can write with greater authority than I on certain phases of the present situation, I shall quote freely from him.

But, on the other hand, it is probable that not many of you have recently read the addresses referred to above, a thing to be strongly recommended at your next opportunity, and it is certainly true that too many of the chemical analysts (not alone the "analytical chemists" in a professional sense) are not putting out a grade of work which is commendable to them. It is not unreasonable, then, that we should take time to survey the field; nor is it true that such a survey will show no signs of improvement from which we may take comfort and courage.

When we consider the relation of analytical chemistry to the other branches of our science, we still face such questions as these: Why is it that analytical chemistry lags behind other branches of chemistry in its scientific development? Why is it that our journals are so often filled with "new" processes of alleged reliability and usefulness, which never acquire general recognition and which are frequently condemned as worthless by those who attempt to apply or repeat them? Why is it that reports of commission after commission on "standard" methods are published, only to sink into oblivion without awakening any interest or attaining any useful purpose? Why is it that the chemical analysts, as a class, lack the respect which should be commanded by those who are following a profession and practising an art which, in its best estate, calls for a very high degree of intelligence, as well as manual skill? Why is it that chemical analyses have come to be made at prices worse than those of the bargain counter? These are, it appears, all pertinent questions to-day.

It has been frequently pointed out that analytical chemistry has so long been regarded as the servant of the chemist—referred to by Ostwald as the "hand-maiden" of chemistry—that far too much attention has been directed to obtaining large returns for small wages. We have nearly forgotten that this "hand-maiden" herself has a natural comeliness, and have ignored the fact that even a little more encouragement to her to improve her methods and accomplish her tasks more thoroughly would easily fit her to sit at the family table—a privilege, formerly hers, of which she has long been deprived. It is easy to see, as Dr. Böttger points out anew, how, after the days of Liebig, the relegation of analytical chemistry to a subordinate position came about amid the interest attaching, first, to rapid developments in organic chemistry, and later to those in physical chemistry, and especially through its connection with the phenomenal growth of the industries, which demanded "results" and too often have allowed what appeared to

be "good enough" for a temporary purpose to become a permanent standard of attainment. These latter conditions must, however, always be reckoned with, and it is not my purpose to spend time in deploring them, or in regretting the conditions of the past, but to ask what encouragement may be offered to the analyst of to-day and what the outlook is for the future.

My chief criticism of the situation in the recent past would be this: That the chemical analysts, as a class, have failed to take advantage of their opportunities, and that those intrusted with their training are considerably responsible for this situation. Specifically, I mean that too many practising analysts allow themselves to remain in ignorance of the aids to accurate, intelligent work which that branch of chemistry known as physical chemistry, in a broad sense, has contributed, and that, while our students are usually made familiar at some time with much of this material, it is not coordinated with their training as analysts. We train too much for manipulative skill and analytical facility and do not sufficiently educate toward that critical intelligence which enables a man to test his own work, to view it from the outside, as it were, and will not allow him to regard it as satisfactory until he has not only assured himself with respect to such matters as the purity of his reagents or the stability of his glassware, but has also studied the effect of a variation of individual factors and of the chemical conditions, so far as his time will permit. Obviously, a practising analyst can rarely afford the time to make his methods of analysis the subject of exhaustive investigations, but something far less than this, yet considerably more than is too often done, would have prevented many deplorable happenings, including the publication of many unreliable analytical procedures to the bewilderment of the entire chemical fraternity.

That the analyst who is ambitious to make the most of his privileges to-day is in a position to obtain a larger measure of aid and comfort than formerly is indicated by an instance cited by Dr. Böttger. He contrasts the work of Professor Clemens Winkler upon the atomic weight of nickel with the later work of Professor J. W. Richards, which showed the figure obtained by Winkler to be in error by 0.3 per cent. Winkler was highly skilled in the technique required for the work which he undertook, and possessed special mental aptitude for the task, as Richards himself has testified. It appears, then, reasonable to conclude that Richards, although similarly equipped, succeeded in attaining greater accuracy than Winkler rather because of his greater ability to recognize those factors which would lead to error than because of greater ability to overcome the difficulties after they had been recognized. Richards had at his command a qualitative and, in some cases, a quantitative knowledge of phenomena, unrecognized until recently, which permitted not only the detection of new sources of inaccuracy, but often enabled him to estimate the extent of the errors involved. He made the knowledge of the day serve his keen intellect to its utmost. He took advantage of his privileges.

But perhaps some of you will say, "This is atomic weight work, not analytical chemistry. Those men are ripe scholars and investigators who can command a knowledge of the advances of their science. All this has little to do with me, a busy analyst, or an unripe scholar." That, however, is just where the issue really lies, and it is because so many have thought and still think that a great deal of the accumulated chemical knowledge in the field of general or physical chemistry is "beyond them"

and is something reserved for the use of those with chemical leisure on their hands, that our progress toward better things is so slow. It is also the reason why a great deal of time is wasted on procedures which are almost without value as soon as any one of the particular conditions (often very far from practical conditions) under which they were tested, is altered. It is not at all a difficult matter to obtain an understanding of at least the meaning, for example, of chemical equilibrium and the laws which apparently underlie equilibrium phenomena; yet there are instances of the publication of processes recommending procedures in defiance of these principles, and a great deal of time is wasted in private because of ignorance of them. An interesting and fruitful application of these principles is to be found in connection with the processes of neutralization and hydrolysis, and in selection of suitable indicators for use with acids and bases of varying strength. Modern research has changed this from a haphazard procedure to one of comparative exactness. The principles are not difficult to follow and, if once mastered, can not fail to render any subsequent work in this field more intelligent and, therefore, more pleasurable.

The phenomena of adsorption, with their attendant annoyances to the analyst, have been widely studied. The results are perhaps less positively helpful than in the case of the indicators, but the material is nevertheless well worth attention and study. Colloids—which probably play an often unsuspected part in our analyses for good or ill—must not be overlooked by the well-informed chemist who would best economize his time and energies, and the simple matter of the best way to wash a filter and its contents, long ago pointed out by Ostwald, are not as familiar as they should be. The spectroscope and microscope are more useful than formerly, a system of micro-analysis, both qualitative and quantitative, having been developed with the latter instrument; the ultramicroscope may well open up new lines of study with respect to the formation of precipitates, the existence of colloids or the conditions controlling electrolytic deposition; wash-waters may now sometimes be advantageously tested through their electrical conductivity; the refractometer, the nephelometer, the colorimeter, the centrifuge, are all finding extended usefulness, and it is incumbent upon the analyst to understand these instruments, and their underlying principles thoroughly if he is to fully avail himself of their aid.

It is probable that analytical chemistry has been as directly aided by the increased accuracy of solubility determinations as in any other way; and these have largely been made practicable by the development of the physico-chemical methods of measurement. These determinations have made it possible to judge of the greatest attainable accuracy of a precipitation method, and have also made practicable the use of a correction factor in the case of unavoidable losses. The analyst should no longer content himself with the mere thought that such losses are inevitable until he has definite assurance that data sufficient to permit of a reasonably accurate estimation of these losses are not to be found. The values obtained for the solubilities of the various sulphides, which are much more accurate than those formerly available, have alone done a great deal to enable the working out of a more reliable scheme of qualitative detection of the elements, one which is based on scientific deductions, confirmed by careful experimentation, as, for example, in the separation of the sulphides of arsenic, antimony and tin by hydrochloric acid, the separation of zinc from the metals of the

copper group, the complete precipitation of lead and cadmium as sulphides.

It is so much a matter of common knowledge that the chemical changes upon which analytical procedures rest are far better understood, and therefore controlled, because of the fruitful hypothesis of Arrhenius, that no extended statement of this seems necessary in this connection. It may, however, be wise to point out that there is a certain tendency to assume that this theory and its applications constitute "physical chemistry" and that there is reason for complacency when one has acquired a fair understanding of these points. While the foregoing statements are far from complete with respect to those points at which our modern chemistry and physics touch analytical chemistry, it must be evident that the possibilities for assistance are far wider and, indeed, more important than a mere ionic interpretation of chemical changes. For it must be admitted that our knowledge of ionic changes, even in a qualitative sense, is still inexact with reference to many reactions familiar to the analyst, and that the most useful data are those of a quantitative character, to obtain which we must command a knowledge of the wider field of physical chemistry in a broad sense.

Modern investigation has then, especially in the border land between physics and chemistry, given us new tools for our trade as chemical analysts. But it has done still more for us by showing us what it means to use them, and it is here that, as analysts, we should learn our lesson. It is, I think, fair to say that the one fundamental reason why much of the published work of individuals or commissions fails of effectiveness and permanence and, in part, the reason for the lack of respect in which the chemical analyst and his work are held, is the lack of appreciation on the part of authors of the one-sidedness of the published results, and of the procedures adopted as adequate or as the best obtainable. The pages of our journals are, unfortunately, too largely occupied by descriptions of processes, which are supported by data obtained under but one set of conditions, and applied at most to one or two sorts of materials. These conditions are empirically established and are, in many cases, not such as could be controlled under the necessary routine of analysis of materials of more complex structure. The tests, upon which efficiency is claimed, have often been made with pure materials, and in solutions, the contents of which could be easily adjusted and determined, contrasting in this respect with customary analytical conditions. Many authors of analytical procedures lose sight of the important fact that the success of these procedures, in their own hands, has usually been attained only at the cost of considerable practise and as the result of the observance of a series of, often, apparently minor modifications, which they more or less unconsciously ignore when they describe the process for publication. It is exactly this really inexcusable ignoring of these conditions, on the one hand, and the even more frequent failure to study their influence systematically, on the other hand, which has condemned much of the published material. It is true that the intelligent author is between two fires—on the one hand his desire to help his colleagues, on the other hand, the board of editors who, in these days of many papers, must insist upon reasonable brevity. As one who has seen many manuscrips in this field, may I suggest, especially to the younger authors, that I am sure that, while the editors must insist on the omission, for example, of statements to the effect that it is inadvisable to remove a stirring rod

from a quantitative solution without washing it, and, while they must advise the elimination of tabulated statements of the failures encountered on the road to success, and must, in general, curb a rather natural desire to send a four-page reprint containing two-pages worth of work to one's friends, they will welcome an increased exactness of statement of the controlling conditions of analyses, especially when it appears that these factors have been intelligently studied.

It is too obvious to really need mention, that not all of the published work is faulty, and it is notably true that some of the more recent work is of exceptionally high character and sets an excellent standard toward which analysts in general should strive. Reference will be made to only two well-known publications—those of Noyes, Bray and their associates on the development of a scheme of qualitative analysis on a scientific and exact basis, and the work of Allen, Johnston and Adams on the determination of sulphur as barium sulphate. In both these there has been a systematic study of sources of error and, where apparently unavoidable errors appear, the possibility of correction for them has been carefully investigated as well as the determination of the special conditions under which these errors could be reduced to a minimum. Dr. Bray has stated to me (and this view is confirmed by Dr. Böttger) that possibly the greatest service of physical chemistry is the impetus which the precise methods which have been worked out have given to the more critical study of the sources of error and to their prevention or correction. This, in their opinion, is a greater service than any other single attainment in itself.

It may be admitted that few employed as analysts can hope to obtain so complete an insight into other fields as is possessed by Noyes, Bray, Böttger or Johnston, and their immediate coworkers. But it is not unreasonable to point out that a much less thorough knowledge would be of enormous assistance and would lead to the prevention of another fault in the point of view of most analysts; namely, the overlooking of the element of compensation of errors in their work. Few things would do more to bring up the character of analytical work than a better appreciation of the fallacies involved in "check analyses" or the false sense of confidence in an approximate summation to one hundred per cent. Just here, I believe, our teachers are often at fault. Every student tends to glorify his "check analyses," too often even to the destruction of his professional integrity. Even when there is no question of honesty involved, there is a blind faith in their infallibility, and this is reflected in much of the published work. The idea that two analyses carried out with practically identical weights of sample and equal quantities of reagents may "agree," but be perfectly worthless because of inherent errors, never really seems to penetrate the mind of many men. The fact that so simple a thing as variation in the quantities taken for analysis is a wise precaution in testing a process is also unperceived. I do not mean to say that most, or possibly all, teachers do not point out these matters, but I do mean to say that many students never get a clear perception of them, and still less of the idea that dependence upon compensation of errors in an analytical procedure is quite insecure unless the factors governing the occurrence of these errors are approximately known. I feel sure that these defects in our teaching would be lessened if we were, in general, to pay less attention to teaching a variety of methods, as such, and more to the careful investigation of a

few, on a scale commensurate with even the limited knowledge which a junior or senior really possesses. This should be done with the purpose of enabling him to criticize his own work, to study it for possible errors, their causes, and, so far as practicable, their amounts. He may then be encouraged to criticize the published work of others, for which, by the way, no end of useful material is at hand. In this connection, it may not be out of place to recommend to chemical analysts, teachers and practitioners an annual perusal of Dr. Hillebrand's address referred to above, in which he deals with the shortcomings of analysts with respect to reagents and manipulative methods, which it has not seemed necessary to attempt to touch upon here.

Finally, just a word regarding the status of the analytical chemist, using this term to denote one with whom analytical chemistry is essentially a vocation. There is, I think, no doubt that this is distinctly unsatisfactory. In the minds of the general public, to be sure, a chemist is essentially an analyst but, in the minds of employers, an analyst is too often an inferior grade of chemist who can be readily displaced by a reasonably intelligent boy, and whom boy's wages should satisfy. Our young men who have been chemically educated almost universally begin their professional careers in the technical field in our analytical laboratory. While we wisely admonish them to fulfil the task at hand in such a way as to earn more than they receive, to more than "make good," in the phrase of the day, we also urge them not to be content with such positions as will permanently restrict them to analytical service. This, too, under existing conditions, is no doubt wise. But it would seem that the time has come when employers should recognize that work entrusted to boys who, while possibly capable of carrying out routine operations, have no ability to deal with any of the inevitable complications arising from exceptional conditions, or work entrusted even to men who are underpaid and without the encouragement of future adequate recognition, is likely to lack the requisite quality for the attainment of the best possible results. It may not be practicable to create permanently attractive positions for a large number of strong men, but there should be more of these men in the works laboratory than are now found there. Moreover, while a works laboratory is confessedly not a savory neighbor, the chemists should courageously demand that they should not be relegated to some ill-lighted, half-ventilated and dirty corner, as is too often done. This does not mean that quartered-oak desks, glass shelves or expensive trimmings should be asked for, but merely such quarters as will permit self-respecting men, who take pride in their work, to operate under conditions which permit them to render their best service. Some may say that this is an easy suggestion to make on the part of one not immediately associated with the stress of industrial conditions, but one which it is difficult to carry out. This is frankly admitted, but the belief persists that the analytical chemists owe it to themselves to make an effort to secure for themselves and their work a larger measure of recognition such as this suggestion would involve.

If we consider the current prices for chemical analyses, one is prone to think that in one field, at least, competition has had full play. It were, of course, rank heresy in this good city to suggest restraint, and yet it seems full time that something should be done, lest the day might come when the chemist would have to pay the manufacturer for the privilege of ma-

king analyses for him. How can an analytical chemist hope to maintain his professional standing when his charges for individual analyses—even when done in quantity—are such that, unless he is to count his own time as without value (and sometimes even under such an assumption), he can not possibly do reliable work without an actual financial loss? I do not wish to draw any unpleasant or unfair inferences as to which horn of such a dilemma the commercial analytical chemists choose; for the most of them are, no doubt, trying to make the best of a difficult situation. Unfortunately, I have no specific remedy to propose, but it is all too evident that these conditions tend to belittle this branch of our science, to result in a large output of inferior work, and to create a distrust which spreads unduly. It behooves us all to at least do what we can to bring home to those who are to place dependence upon work done at these ruinous prices that, in many instances, they are getting just about what they pay for and no more, and that the service is not such as they owe it to themselves to make possible by more adequate remuneration.

Whether we consider present analytical practise from the view of the scientist or as a vocation, we find much that calls for improvement. What the present situation imperatively demands, then, is a courageous and frank admission that the quality of much of the analytical work, practised or published, is inferior to what might reasonably be attained because much of it is one-sided and ill-considered from a scientific standpoint. Let there be a realization that, while no amount of theorizing can take the place of skilful and accurate work, or of a certain amount of empirical experimentation, the analysts should cease pulling on their own boot-straps and avail themselves more generally of the aids from other portions of our science to help to lift themselves and their art to the worthy position to which both are entitled. Let the chemical analysts realize that they must take greater pride in their work for its own sake, let them demand a recognition of the dignity claimed for it by Dr. Dudley, and let them give to it the best that is in them, in both activity of mind and skill of hand. This is a duty which is owed to our national reputation, to chemical science, pure and applied, and to our own welfare.

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The Crocker Land Expedition under the Auspices of the American Museum of Natural History and the American Geographical Society

The existence or non-existence of land northwest of Grant Land and the configuration of the polar continental shelf of North America seem to be two of the greatest of the geographical problems still unsolved. There is, in addition, much important geological, geographical, zoological and other scientific work to be done in certain parts of the Arctic regions. Two thoroughly qualified young men have volunteered their services as leaders of an expedition to attack the problems and do the work. Under this combination of circumstances and with the proviso that sufficient funds be provided from outside sources, the American Museum of Natural History and the American Geographical Society have made liberal appropriations in support of the enterprise, and the former institution has taken over its organization and management, feeling that it is well worthy of the backing of the scientific institutions of the country.

The leaders of the expedition are to be George Borup (A.B., Yale, 1907), assistant curator of geology in the American Museum